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विभाजन की विधि
(पहला पुनरीक्षण)

**Particulate Materials — Sampling
and Sample Splitting Technique**
(*First Revision*)

ICS 19.120

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional Committee had been approved by the Civil Engineering Division Council.

The characterization of particle properties like size, form and specific surface area requires very careful sampling and sample` splitting practices to be followed. The distributions of the values of such properties are related to the number of particles, which cannot be increased as in sampling for chemical analysis. Deviations from statistical values occur due to the presence of particles of different sizes and shapes for each component in a powder obtained from a sampled mass of powder.

This standard was first formulated in 1979 with the title ‘Method of sub-division of gross sample of powder used for determination of particle size’. It covered different methods of sub-division of gross sample down to required quantity for further analysis for determination of particle size distribution. In this revision, the standard has been updated in line with ISO 14488 : 2007 ‘Particulate materials — Sampling and sample splitting for the determination of particulate properties’. It now covers detailed description of principles of sampling and sample splitting, sampling plan, various division techniques and validation, apart from other details.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PARTICULATE MATERIALS — SAMPLING AND SAMPLE SPLITTING TECHNIQUE

(First Revision)

1 SCOPE

This standard specifies methods for obtaining a test sample from a defined bulk of particulate material (powder, paste, suspension or dust) that can be considered to be representative of that bulk with a defined confidence level. It is particularly relevant to the measurement of particle size, size distribution and surface area.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	Title
IS 101 (Part 10/ Sec 1) : 2022/ ISO 13320 : 2022	Methods of sampling and test for paints, varnishes and related products: Part 10 Instrumental analysis, Section 1 Particle size analysis — Laser diffraction methods (<i>first revision</i>)
IS 8883 (Part 1) : 2005	Methods of sampling chemical and chemical product: Part 1 General requirements and precautions (<i>first revision</i>)
IS 18100 : 2022	Sample preparation — Dispersing procedures for powders in liquids

3 TERMS AND DEFINITIONS

For the purposes of this standard, the terms and definitions given in IS 8883 (Part 1) and the following apply.

3.1 Analysis Sample — The portion of the laboratory sample which is used in the size analysis apparatus.

3.2 Bias — Systematic difference between true (or accepted) value and measured value.

3.3 Critical Size Class — Specific size class, whose sampling error, in its fractional mass, has a significant influence upon the product properties.

3.4 Error — Difference between a measured value and the true value, which may have a random or a systematic nature.

3.5 Grab Sample — Sample that has not been taken under well-defined conditions.

3.6 Gross Sample — Primary sample, composed of several sample increments.

3.7 Laboratory Sample — The portion of the gross sample which is delivered to the laboratory for determination of particle size distribution.

3.8 Primary Sample — Sample (single or composed) taken from a defined bulk product.

3.9 Representative Sample — Sample that has the same properties as a defined batch of material and represents the bulk material, within a defined confidence limit.

3.10 Sample — Part of a defined bulk product taken for the purpose of characterization.

3.11 Sample Increment — Single sample, taken from any of a defined set of locations in a bulk product or at any of a defined set of times from a production/transportation line, to be mixed with other increments to form a gross sample.

3.12 Sampling Sequence — Sequence of sampling, sample division and combination steps that result in a test sample for a defined bulk product.

3.13 Spot Sample — Sample, taken at a defined location or production time, from a batch of material.

3.14 Test Sample — Sample that is entirely used for a property characterization.

4 PRINCIPLES OF SAMPLING AND SAMPLE SPLITTING**4.1 General**

Particulate materials consist of discrete particles, each having its own properties such as size, shape, surface area, density and/or composition. Sometimes, the material is well mixed and the properties show only random variations with respect to location in the bulk and/or time of production. More often, however, segregation occurs due to the free-flowing behaviour of the material and/or fluctuations in the production

process. This can result in a systematic deviation between the mean properties at different locations and at different times. For representative sampling, each of the particles in a bulk product must have the same probability of being sampled, in their proportions. For well-mixed materials, a single sample of adequate quantity may suffice. For most materials, some degree of segregation is to be expected. Then, several sample increments must be taken from different locations or at different production times. These are either analyzed as such or combined into one primary sample. In most cases, there is no recipe for representative sampling. The quality of the sampling procedure can only be assessed by measurement. Often, the primary sample collected in this way is too large for characterization of the required property. Then, sample splitting is necessary until the required quantity for the test is obtained.

The total error of sampling and sample splitting consists of two parts:

- a) Fundamental error; and
- b) Segregation error.

The fundamental error is related to the discrete nature of particles given their different properties. It is a statistical error, related to the random variations of the properties with respect to location. It represents the lower limit of the total sampling error. This error depends on the amount (number, mass) of sample taken.

The segregation error is related to the degree of segregation, or the degree of 'de-mixing' of particles according to their size, shape and density. It cannot be predicted, but only assessed by measurement of samples taken at different locations in the bulk and at different times of production. These measurements provide an estimation of the segregation error (see 4.3).

Given the complex behaviour of particulate materials, it is required that the complete procedure for sampling and sample splitting be described in a sampling plan (see 5).

4.2 Fundamental Error

4.2.1 Number Distributions $Q_0(x)$

For number-based size distributions, the fundamental error [expressed as the variance Var or squared standard deviation σ of a fraction $Q_0(x_i)$ at any point x_i] can be calculated using the mathematics of binomial distributions:

$$\text{Var}[Q_0(x_i)] = \sigma_{Q_0,i}^2 = [1 - Q_0(x_i)]/n \quad \dots (1)$$

If the number of particles in a size class or above a certain size is very small in comparison to the

total number of particles taken into account for a measurement, then Poisson statistics may be used for estimating the variance or the standard deviation:

$$\text{Var}(n_0) = \sigma(n_0)^2 = n_0 \quad \dots (2)$$

For example, for the number of particles larger than x_{90} , it can be seen that

$$n_0 = [1 - Q_0(x_{90})]n \quad \dots (3)$$

and, since $Q_0(x_{90}) = 0.9$ or almost 1, Equations (1) and (3) give approximately the same answers.

where

- Var = variance;
- $Q_0(x_i)$ = cumulative number-based particle size distribution;
- σ = standard deviation; square root of variance (theoretical value);
- n = total number of particles in sample or sample increment;
- n_0 = number of particles in a defined size class;
- x_{95} = 95 percentile size of the particles;
- x_5 = 5 percentile size of the particles; and
- x = particle size.

If the fundamental error is the only error, the minimum number of particles $n_{\min}$ required to obtain a defined maximum deviation ε_{Max} with a defined confidence can be derived from:

$$\varepsilon_{\text{Max}} = z_c \sigma_{Q_0,i} = z_c \sqrt{Q_0(x_i)[1 - Q_0(x_i)]/n_{\text{Max}}} \quad \dots (4)$$

or

$$n_{\text{Min}} = \{Q_0(x_i)[1 - Q_0(x_i)]\} z_c^{2/\varepsilon_{\text{Max}}^2} \quad \dots (5)$$

The standard deviation of x_i can be calculated from $\sigma_{Q_0,i}$ through multiplication by the reciprocal value of the slope of the cumulative size distribution at point x_i :

$$\sigma_{x_i} = \sigma_{Q_0,i} dx_i/dQ_0(x_i) \quad \dots (6)$$

The coefficient of variation of x_i can be calculated from this standard deviation by multiplication by 100 and division by x_i :

$$\text{CV}_{x_i} = 100 \sigma_{x_i}/x_i \quad \dots (7)$$

where

- ε_{Max} = defined maximum level of deviation at defined confidence level (half-width of the stated confidence interval);

- z_c = critical y -value related to a defined confidence level according to the Standard normal distribution (to be taken from statistical tables);
- n_{Min} = number of particles in a sample or sample increment required to obtain a defined maximum deviation ε_{Max} with a defined level of confidence;
- CV = coefficient of variation, that is, standard deviation relative to the corresponding mean value, expressed as fraction or percentage; and
- x_i = particles size corresponding to percentile i .

4.2.2 Volume or Mass-Based Distributions $Q_3(x)$

For volume or mass-based size distributions, the general calculation of the fundamental error is not simple.

Convert the measured volume-based size distribution of a typical sample into the corresponding number distribution.

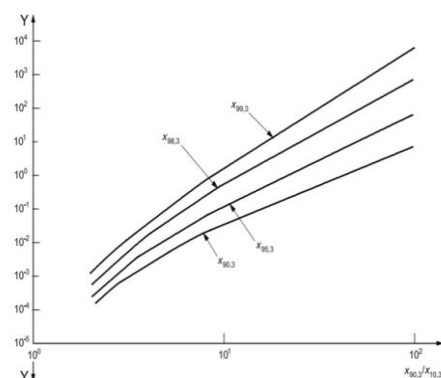
This estimation of the minimum amount of sample in view of a stated minimum fundamental error shall always be the first step in a sampling procedure.

As an example of the results from such calculations, Fig. 1 and Fig. 2 are presented for a 1 percent coefficient of variation in x for various characteristic sizes as log-normal size distributions. The calculations were conducted for distributions around a median size of $30\ \mu\text{m}$ with a material density of $1\ 000\ \text{kg/m}^3$. A detailed calculation is given in Annex A.

Figure 1 shows that, for a constant percentile, the sample mass required to reach a coefficient of variation of 1 percent increases with particle size distribution width. It increases from about 10 mg to 1 kg as the distribution width ratio ($x_{90,3}/x_{10,3}$) increases from 2 to 100. The sample mass required is also increased if the fundamental error is to be maintained for percentiles ever closer to the highest limit of the distribution ($x_{90,3}$, $x_{95,3}$, $x_{98,3}$ and $x_{99,3}$). For a distribution by mass, it should be noted that a 10 times higher mean particle size requires a 1 000 times higher sample mass and *vice versa*. Note also that acceptance of a 10 times larger coefficient of variation in the fundamental error requires 100 times less sample mass.

Figure 2 illustrates that, for larger distribution width ratios, larger numbers of particles must be counted to ensure a result whose error remains below the target of a 1 percent coefficient of

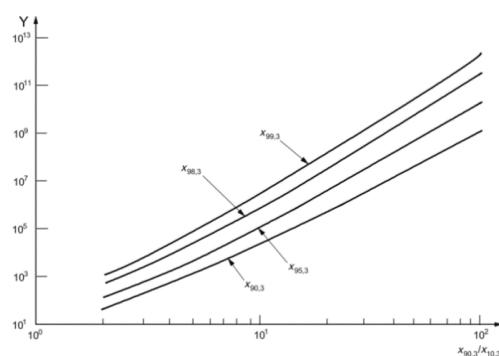
variation. For a very narrow size distribution, counting a few hundred particles is all that is required. However, for very broad distributions, a number in excess of 10 000 000 will need to be assessed and counted.



Key

Y Minimum sample mass (g)

FIG. 1 MINIMUM SAMPLE MASS FOR 1 PERCENT FE



Key

Y Ratio of total number of particles to the number larger than the specified size

FIG. 2 RATIO OF TOTAL TO FRACTIONAL PARTICLE NUMBER

4.3 Total Error/Number of Samples or Increments

It is to be expected that some degree of segregation will have occurred in the bulk product. The total sampling error shall be assessed through measurements of the property of interest in multiple samples. These samples shall have about the same size and come from different locations in the bulk or have been taken at different times during the production process. The minimum sample size shall be calculated as indicated in 4.2 and Annex A. From these multiple measurements of the value y of a property, both the mean value $\bar{y}$ and its standard deviation s_y may be calculated from:

$$\bar{y} = \frac{1}{n} \sum y_i \quad \dots (8)$$

and

$$s_y = \sqrt{\frac{\sum_i (\bar{y} - y_i)^2}{N-1}} \quad \dots (9)$$

This total error may first be compared with the fundamental error as calculated above and the degree of segregation assessed. Secondly, it can be converted to a confidence interval indicating a maximum level of deviation ε_{Max} at a given level of confidence using

$$\varepsilon_{Max} = t s_y / \sqrt{N} \quad \dots (10)$$

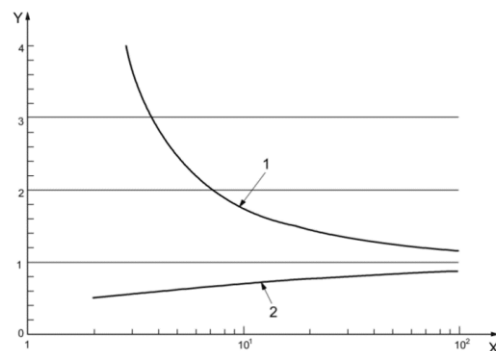
Equation (10) may be rearranged to calculate the number of samples (or sample increments) N_r required for a deviation ε_{max} at a defined level of confidence between the estimated and the true mean value of y .

$$N_r = (t s_y / \varepsilon_{Max})^2 \quad \dots (11)$$

where

- $\bar{y}$ = mean value of y ;
- s_y = estimate of standard deviation of y , coming from measurements;
- t = student's t – factor for statistical significance, which depends on the confidence level taken and the number of degrees of freedom ($N - 1$) (to be taken from statistical tables);
- t -value = it is dependent upon the confidence level taken and the number of degrees off freedom ($N - 1$);
- N = number of measured samples;
- N_r = number of samples required to obtain a defined maximum deviation ε_{Max} , with a defined level of confidence, between the estimated and the true mean value of a property of interest; and
- FE = fundamental error.

When the measured values y of a property follow a normal Gaussian distribution, which is generally true, then the confidence interval of the variance of these measurements can be derived from a chi-squared distribution, which is not symmetrical. This is illustrated in Fig. 3, which shows the 95 percent confidence limits for the ratio of true standard deviation to calculated standard deviation as a function of the number of degrees of freedom.



Key

- X Number of degrees of freedom
- Y Ratio of true standard deviation to calculated standard deviation
- 1 Upper limit
- 2 Lower limit

FIG. 3 95 PERCENT CONFIDENCE LIMITS FOR CONFIDENCE INTERVAL OF STANDARD DEVIATION

Figure 3 shows stabilization of this confidence interval when generally more than 30 samples are taken into account for the best estimate of precision.

When measuring particle size and surface area, the exercise of estimating the standard deviation of the mean measured property is only performed at the beginning, when starting with a given bulk product or production process, or during validation. When products are to be checked on a regular basis, the minimum number of samples required to assess the product quality may be taken as sample increments. These sample increments may be combined to create a gross sample if they have the same mass. Such a gross sample may be too large for characterization and thus has to be subdivided to obtain the required quantity of test sample. During subdivision (sample splitting), the same equations can be applied as indicated earlier in this clause.

5 SAMPLING PLAN

A sampling plan shall be specified and documented. The sampling plan shall consist of a general part and a technical part.

It is recommended, but not mandatory, that the sampling plan to be made for the whole sampling sequence from primary sampling to the final test portions.

The general part shall address at least the following aspects:

- a) Material description;
- b) All known relevant information on the powder to be measured (for example,

- variance, hygroscopicity, reactivity, cohesion, etc);
- c) All known hazards related to the powder (for example, toxicity, flammability);
- d) Description of the task;
- e) The use of the information to be estimated from the powder (for example, sales contract, internal quality measurement);
- f) The random variables to be estimated in exact terms (for example, mean percentage of material finer than 10 µm in production batch); and
- g) The variance (coefficient of variation) and the related limit of confidence shall be specified for all sample splitting stages. The total sample splitting variance estimate shall also be computed.

The technical part shall address at least the following aspects:

- a) Physical appearance of powder at sampling points;
- b) Sampling sites for all sampling and sample splitting stages, including site requirements;
- c) Method of primary sampling, if applicable (random, systematic, stratified, staged, sequential);
- d) Mode of increment withdrawal (continuous, discrete);
- e) Type of increment (time, mass, number).
- f) Size of increments (number of particles or mass) in sampling and sample splitting stages;
- g) Number of increments to be taken in primary sampling and subsequent sample splitting stages;
- h) Number of increments to be chosen for further sample splitting in each stage;
- j) Combination of increments (if applicable);
- k) Required reduction ratios for all sample splitting stages and overall reduction ratio for test sample;
- m) Method(s) of sample splitting (one or more stages);
- n) Detailed description of all other sample handling procedures used (that is, mixing, sonification, dispersant additions, etc);
- p) Method of final analysis;
- q) Number of measurement repetitions for each test sample;
- r) All equipment needed for sampling and sample splitting stages;
- s) Cleaning procedures for all equipment and sample containers;

- t) Qualifications of personnel for each stage of the sampling sequence;
- u) Sample marking; and
- w) Sample storage procedure(s) and place(s).

All equipment and methods shall be validated and such documents shall be a part of the sampling plan. The requirements specified in 6 shall be applied.

6 GENERAL PROCEDURES

6.1 Safety Precautions

Safety precautions are necessary when sampling and handling hazardous materials. Relevant statutory requirements shall be followed and reference shall be made in the sampling plan to any toxicological data, precautionary measures to protect the operator and remedial measures in the event of an accident.

6.2 Primary Sampling

Representative sampling from a bulk particulate product requires that all particles have equal probability of being taken into the sample, in their given proportions. Sample increments of about the same size shall be taken from as many locations in the bulk product as practical and combined into a single gross sample. These locations shall be chosen carefully and be described in the sampling plan. In order to minimize errors due to segregation, this sampling shall be done at a place where segregation is minimal. Sampling from a moving stream is a preferred method. Some general rules for designing sampling equipment are as follows:

- a) The preferred design is one which collects the sample over the whole particulate stream for part of the time and not a fixed part of the stream continuously;
- b) The frequency of sampling shall differ from that of any major process fluctuations or loading sequences. If the frequency of such fluctuations is unknown, further investigations are necessary;
- c) The sampler and sample container shall have no constraints, so that even the largest particles are easily accepted. Thus, the diameter of sampling tubes and the distance between cutter edges shall be at least three times the size of the largest particle and not be smaller than 10 mm. However, for liquid dispersions of small particles, a minimum size of 2 mm is acceptable;

- d) The sample container shall have sufficient volume to easily receive the full sample;
- e) Samplers shall have thin walls or sharp edges to promote good control of acceptance of particles based on their centre of gravity;
- f) The speed of a sample cutter or sample tube shall be sufficiently low in order to avoid large particles being lost by collision with the sides of the cutter or tube and small particles being carried away in the upward air stream. As a rule of thumb, cutter speed shall be lower than 40 cm/s;
- g) Local sampling tubes in pipelines shall be iso-axial to the pipe; and
- h) Local sampling in pipelines shall be done iso-kinetically where possible, while keeping the linear flow rate about the same. The flow in the pipeline shall be either undisturbed or mixed thoroughly. No intermediate flow regimes shall be used.

NOTE — Manual sampling from heaps always leads, with segregating materials, to large sampling errors.

6.3 Sample Handling

Sample increments shall be entirely utilized at all stages of sample division, preparation and measurement. No material may be added to or taken from the increment. Incomplete or contaminated increments shall not be used.

Throughout all the procedures and stages, the sample shall be protected from any contamination by foreign particles.

Samples shall not be unduly transported or handled. If transportation cannot be avoided, however, the method of transportation shall be agreed upon between the parties concerned. Transportation and handling shall be validated to ensure that changes in the particle properties of interest do not occur.

Any sample handling procedures, such as homogenization by mixing, thermal drying or dispersion by sonication or other methods, shall be validated to ensure that changes in the particle properties of interest do not occur.

Appropriate and validated thermal drying conditions (heat source, temperature, atmosphere, ventilation, etc) shall be specified. After thermal drying, the material shall be allowed to cool in a desiccator. If the material is hygroscopic, it shall also be stored in a desiccator.

Appropriate and validated dispersion conditions shall be specified.

Samples shall be stored so that no alteration in the properties of interest takes place. They shall be stored away from direct daylight and shall not be subject to large temperature changes. Longer periods of storage shall be validated.

Any equipment needed in the process of sampling shall be thoroughly cleaned.

6.4 Sample Containers

Storage containers used for storing any sample shall be capable of holding the entire quantity of the sample. The storage container shall be clean and sufficiently robust. The storage container shall either have a lid to close it securely or be of such construction that it can be closed tightly in another way.

6.5 Marking of Sample Containers

Containers shall be marked if the divided sample is not going to be used immediately. The container markings shall include the following information:

- a) Sample identification:
 - 1) Number, and
 - 2) In the case of parallel samples, the total number of samples and the sequence number within the total.
- b) Sampling level identification;
- c) Name of commodity;
- d) Identification of lot number and producer's name;
- e) Date of sample preparation;
- f) Name of person who carried out the sample preparation and the affiliation; and
- g) Other necessary information (if applicable):
 - 1) Carrier fluid identification,
 - 2) Any dispersion and stabilization reagents employed,
 - 3) Other treatments to which the sample has been subjected,
 - 4) Special properties of the material, such as hygroscopicity, segregation tendency,
 - 5) Information concerning safety, and
 - 6) Identification of the sampling plan applied.

An alternate strategy is to mark the sample containers with a sample identification number which can be related to the above information recorded in a sample preparation laboratory book or computer file. Computer files shall have a backup. In the event of further division of a stored sample, the original sample identification shall be recorded on the containers containing the newly divided samples. Markings on sample containers shall be made in such a way that they will not become illegible during the storage period.

7 SAMPLE DIVISION TECHNIQUES

7.1 General

Often the size of primary samples is too large for property characterization purposes. In such cases, the sample shall be divided to give a test sample of a suitable size. Several techniques exist for sample splitting. Their applicability depends on the flow properties and the homogeneity of the primary sample from which a representative test sample is desired. Sometimes, different techniques are used in combination.

7.2 Spinning Riffler

Spinning rifflers (rotary samplers) are the best choice to sub-sample free-flowing particulate materials. Other materials may be made to flow freely by the use of additional agents in known quantities. Sticky and strongly agglomerated powders are excluded. Spinning rifflers are available in different sizes; they can provide samples ranging from about 0.5 g to 300 g.

The spinning riffler consists of a feed hopper, a vibratory feeder and a turntable or other rotating divider ring where the sample holders are located.

Before sample splitting, the sampler shall be clean. The hopper design shall be such that a constant, non-segregating, even mass flow of sampled material is ensured. The sample holders shall be designed in such a way that no material to be sampled is lost outside the sample holders or remains trapped within the hopper, vibratory feeder, divider ring or any other part of the sampler. Special care shall be taken in the riffler design and its operation that material will not be retained between the sample holders. The material shall be carefully mixed before being placed in the feed hopper. The vibratory feeder is then actuated and left to operate until the entire sample has been divided. The hopper and the vibratory feeder shall be cleaned while the riffler is still running so that no material remains in the hopper or in the vibratory feeder.

7.3 Static Riffle Divider

Static rifflers (table rifflers or chute rifflers) are capable of providing large quantities of samples for materials that are free-flowing or can be made to flow evenly. Their quality of division is generally worse than that of spinning rifflers. They usually find application with very coarse materials. They shall not be used for materials that readily liberate dust, nor for friable materials.

The material shall be carefully mixed in a feed container and poured uniformly into the divider to divide the sample into parts. Any one of the

divided parts shall be selected randomly as the divided sample. Care shall be taken to prevent clogging of the riffles. If clogging does occur, the whole sample shall be re-mixed and a new pour carried out after cleaning the riffler.

Slow pouring of sample into the riffler shall be avoided.

The size of the riffle divider shall be in accordance with Table 1.

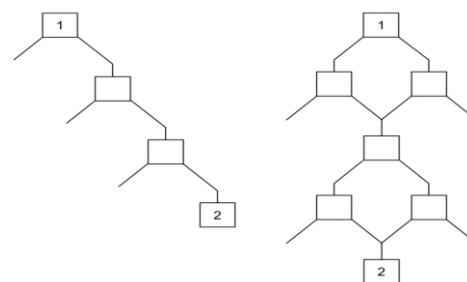
Table 1 Size of Static Riffler to be used

(Clause 7.3)

SI No.	Maximum Particle Size	Opening Width
	mm	mm
(1)	(2)	(3)
i)	20 to 16	50
ii)	16 to 10	30
iii)	10 to 5	20
iv)	5 to 2.5	10
v)	below 2.5	6

The static riffler shall be used in accordance with one of the two methods illustrated in Fig. 4.

In method 1, the sample is divided into two parts. One of these parts is further divided into two, and so on, until the sample size required for final analysis is obtained. The reduction ratio for division into l stages is $\frac{1}{2^l}$.



Key

- 1 Sample to be divided
- 2 Divided sample

FIG. 4 POURING SCHEMES FOR A RIFFLER

In method 2, the sample is divided into two parts and both parts are separately divided into two. Two of the halves produced by this division are combined and the resulting combined sample then divided again in the same two-step mode.

The reduction ratio is $\frac{1}{2^{l/3}}$

Method 2 is the preferred method.

7.4 Coning and Quartering

7.4.1 General

The technique of coning and quartering can be applied successfully for powders with poor flow properties. Due to the manual operations involved, the technique tends to be operator sensitive.

7.4.2 Coning and Quartering of Powder

The material to be coned and quartered shall be thoroughly mixed in a mixing container. The material is then poured carefully on to a hard, clean, nonporous surface. The pouring shall be made in a single pass and the pouring point (spatial location) shall not be moved from the centreline of the cone which forms during the pour.

For quartering, a knife with four crossed blades is used. The height of each blade shall be more than the height of the sample cone formed by pouring. The knife is carefully inserted into the top of the sample. The movement of the blade shall follow the centreline of the sample cone. Care shall be taken that the knife is inserted right to the bottom of the cone. A randomly chosen quarter is then removed as a sample without moving the knife. All the particles shall be removed, using a small brush if necessary.

This method shall not be used for powders having a tendency to segregate due to large differences in density or in particle size, or for powders that are prone to dust liberation. The method shall not be used for hazardous materials.

NOTE — This method tends to give a high random error and is operator sensitive. It may also give systematic errors. It shall be validated before use.

7.4.3 Coning and Quartering of a Paste

When using the coning and quartering method for pastes, form the material into a cone shape. A conical mould in two halves may be used. Otherwise, the procedure is similar to the coning and quartering of dry powders.

Direct quartering of a filter cake shall not be carried out.

7.5 Increment Division Method

The increment division method can be used not only for materials with good flow properties but also for those with poor flow properties. Here, too, the quality of sample division depends upon the ability of the operator.

The material shall first be mixed thoroughly without dust formation. The material is then spread on a flat, clean surface and formed into a rectangular bed having a uniform thickness as given in Table 2. Mark the rectangle into areas of

equal size, the number of areas corresponding to the number of increments required. Take a sample from each area, selecting the sampling point within the area randomly. Before sampling, insert a plate, against which the sample will be taken, from the top to the bottom of the powder layer. Then, insert a sampling shovel into the sample layer down to the bottom of the layer. Use a blunt-ended shovel with sidewalls. Take the sample in a direction perpendicular to the inserted plate, advancing the shovel until it makes contact with the plate. When removing the shovel from the sample bed, remove the plate and shovel together to ensure that no material is lost. Combine the sub-samples thus obtained to give a combined sample.

Table 2 Bed Thickness in Manual Incremental Division of Sample
(Clause 7.5)

SI No.	Maximum Particle Size mm	Thickness of Sample Layer mm
(1)	(2)	(3)
i)	20	35 to 60
ii)	16	30 to 45
iii)	8	25 to 35
iv)	4	20 to 30
v)	2	15 to 25
vi)	1	10 to 15
vii)	0.5 or finer	5 to 10

If a different quantity of sample is needed than that obtained by the above procedure, either the size of the increment or the number of increments shall be varied. By weighing the first sub-sample obtained, an estimate of the size of the final combined sample can be computed. If adjustments are necessary, the sub-sample already taken is returned to the original material and the procedure is re-started with a new increment size or number of increments.

This method shall not be used for powders having a tendency to segregate. Nor shall it be used for materials that show a large variation in particle density or size or for materials that are prone to dust liberation. The method shall not be used for hazardous materials.

NOTE — This method tends to give a high random error and is operator sensitive. It may also give systematic errors. The method shall be validated before use.

7.6 Scoop Sampling

Scoop sampling is, in general, subject to large errors. Only for well-mixed, sticky powders having a fairly narrow particle size distribution, or

for pastes, might it provide adequate quality. The technique tends to be very operator sensitive due to the manual nature of the operations involved.

Before sampling, the particulate material shall be mixed well within its container by stirring or rotation. Free-flowing materials shall be mixed with a few drops of liquid, containing some dispersant, to form a paste (*see 7.7*). Several sample increments, preferably at least ten, shall be taken by means of a scoop, spoon or spatula and combined to form the test sample.

7.7 Sampling from Paste

This method allows extraction of very small samples from pastes and powders after they have been mixed into a (homogeneous) paste. In a paste, the high concentration of solids in the liquid restricts segregation of particles.

Small spot samples to be used as test portions may be obtained from a well-mixed paste made of fine powder.

Prior to preparing the paste, prepare a dispersing liquid containing suitable amounts of dispersants. Care shall be taken that the dispersing liquid contains no dispersant particles that may interfere with the measurement.

Place a weighed amount of solids onto a clock glass. Add a few drops of the prepared dispersing liquid and carefully knead the powder to a paste. The total amount of liquid shall be such that the material is barely in paste form. Care shall therefore be taken in adding the final drops of dispersing liquid.

After the paste has formed, it shall be thoroughly mixed.

Samples may be taken from the paste either at random or systematically. In systematic sampling, the surface of the paste is divided into parts and a sample is taken from each part.

This method shall be validated by comparing it with another sample splitting method to confirm that no particle breakage or dissolution occurs.

7.8 Suspension Sampling

7.8.1 General

Suspension sampling allows preparation of very small samples (10 cm³ or less) from suspensions and also from powders after they have been mixed with a dispersant to give a homogeneous suspension. The suspension shall, of course, be

stable (no sedimentation) during the period of sampling.

7.8.2 Sample Splitting by Pipette

With powders, prepare the suspension by filling a cylindrical vessel with liquid containing a suitable amount of dispersant and add the sub-sample prepared by any of the methods described earlier. Dispersing procedures shall be in accordance with IS 18100 'Sample preparation — Dispersing procedures for powders in liquids'. Take care that the powder is properly wetted and ensure that no solids are left on the walls of the vessel. The vessel shall be equipped with a stirrer and with baffles to prevent symmetrical flow during mixing. Agitate the suspension thoroughly and, while agitating, withdraw aliquots containing the required test portion amounts using a pipette or dropping tube of an appropriate volume and orifice diameter.

After sampling, flush the pipette with particle-free suspension liquid and add this liquid to the sample aliquot taken.

Before reinserting the pipette in the suspension, clean the outside of the pipette.

This method is best suited for powders with a maximum particle size of about 20 µm to 40 µm. It is not well suited for powders having a large variation in particle density, shape or size.

NOTE — This method may cause systematically low contents of coarse particles if used for powders containing coarse and/or dense particles.

The method shall be validated for all new materials.

7.8.3 Sample Splitting Using Multiple Capillary Tubes

Multiple suspension samples may be obtained simultaneously by permitting a dispersed sample to flow through a set of capillary tubes spaced symmetrically in a vessel. Typically, the capillary tubes have a bore of 0.7 mm. The sampling process may be validated by measuring the volume of particle-free suspension liquid which comes through each capillary. The volume delivered by the capillaries to the sample receivers shall be accurate to within 2 percent.

Check that the suspension sampler is clean. Test the sampler, both before and after the sample splitting operation, for blocked capillary tubes. To do this, add particle-free suspension liquid to the sampler and observe the flow of liquid from the capillaries while applying an over pressure to the sampler. If any blockage is observed, clean the blocked capillaries and repeat the check.

It is recommended that the sampler not be used for powders whose maximum particle size exceeds about one-third of the capillary tube bore.

Prepare a suitable amount (about 100 cm³) of suspension in a separate container and pour it into a glass vessel of about 150 cm³ which can be tightly sealed and is equipped with a stirrer and capillary tubes attached to its base plate. Wash out the container, adding the washing liquid to the suspension in the glass vessel. Make sure that the sample receivers can receive the whole of the prepared suspension plus all the washing liquid. Mix the suspension thoroughly for 15 s to 30 s. While mixing, apply a slight under-pressure to the sampler to keep the capillaries free of suspension. Place the capillary tubes in the sample receivers and, while continuing to stir the suspension, split the sample by applying an over pressure to the sampler. When splitting is complete, thoroughly wash the sampler.

8 VALIDATION

The quality of the primary sampling procedure shall be validated using the actual material and method adopted. The standard deviation and confidence interval shall be calculated using the procedures given in 4.3 and the maximum error at

the given confidence level shall be compared with the required maximum error.

The quality of the sample splitting technique(s) shall also be validated.

The simplest validation to be performed when the dry riffler splitting technique is used is a mass validation.

Measure the mass of the gross sample together with the masses of all the increments. Repeat this three times. If the mean loss of material is larger than 1 percent, or if the loss in any single experiment is more than 1.3 percent, the riffler is deemed to be not working properly and this shall be corrected before further use. If the coefficient of variation in the mass of the increments is larger than 1 percent, then the riffler is also deemed to be not working properly.

For a more rigorous validation of the sample splitting method, the method given in Annex B can be used. The material to be split shall be a standard reference material that has a similar particle size distribution and similar flow ability to the material to be treated.

The results shall be verified against the required total variance. The results can also be used to design the sample treatment chain.

ANNEX A

(Clauses 4.2.2 and 4.3)

ESTIMATION OF SAMPLING ERRORS AND MINIMUM MASS OF SAMPLE

A-1 GENERAL

This annex provides examples of how to estimate sampling errors and the minimum mass of sample required for a stated precision. Generally, the total sampling error consists of two parts: a fundamental error (FE), which is due to the particulate nature of the material, and a segregation error, which is caused by segregation of the material. In **A-2** of this annex, a graph is presented which relates, in the case of a log-normal particle size distribution, the sample mass required for a 3 percent fundamental error in the quantity of material larger than $x_{90,3}$ to the median particle size and the width of the distribution. **A-3** gives an example of the calculation of the sample mass needed for a 3 percent fundamental error when sampling glass beads. **A-4** converts a fundamental error in the quantity of oversized material into that for the corresponding characteristic size. **A-5** presents an example of the calculation of the total sampling error from a series of measurements. **A-6** summarizes a procedure for estimating the minimum amount of sample (increment) and the minimum number of samples/increments needed to reach a given precision for a given value in a particle size distribution.

A-2 GRAPHICAL REPRESENTATION OF SAMPLE MASS REQUIRED FOR A FUNDAMENTAL ERROR OF 3 PERCENT

In Fig. 5, the lines indicate, for a log-normal size distribution, the sample mass required to reach a fundamental error of 3 percent in a quantity of material larger than $x_{90,3}$ in relation to the median size and the width of the distribution. The width of the distribution is expressed as the ratio between $x_{90,3}$ and $x_{10,3}$, and the material is assumed to have a true density of 1 000 kg/m³. Fig. 5 gives a quick indication of the sample mass required to obtain an FE of 3 percent in the amount above $x_{90,3}$, provided that an adequate estimate of the particle size distribution is available.

Figure 5 also illustrates the considerable increase in

the required sample mass as the particle size and/or the width of the size distribution increases. In the sub-micrometre region, the minimum sample mass is about 1 µg; for millimetre-sized particles it is about 0.1 kg to 1 kg. For materials having a true density higher than 1 000 kg/m³, the sample mass has to be multiplied by the known particle density divided by 1 000.

A-3 EXAMPLE OF THE CALCULATION OF THE SAMPLE MASS NEEDED TO OBTAIN A 3 PERCENT FE WHEN SAMPLING GLASS BEADS

This example illustrates a procedure for the calculation of the sample mass required to reach a stated precision for the FE in a quantity of material larger than a stated characteristic size in a size distribution. The example uses a precision (coefficient of variation) of 3 percent for the fundamental error and $x_{90,3}$ as the characteristic size. Other values may be chosen.

It is assumed that the variance of the fundamental error and the number of particles n_0 are related in accordance with Poisson statistics [see Equation (2)]:

$$\text{Var}(n_0) = \sigma(n_0)^2 = n_0$$

$$\sigma(n_0) = \sqrt{(n_0)}$$

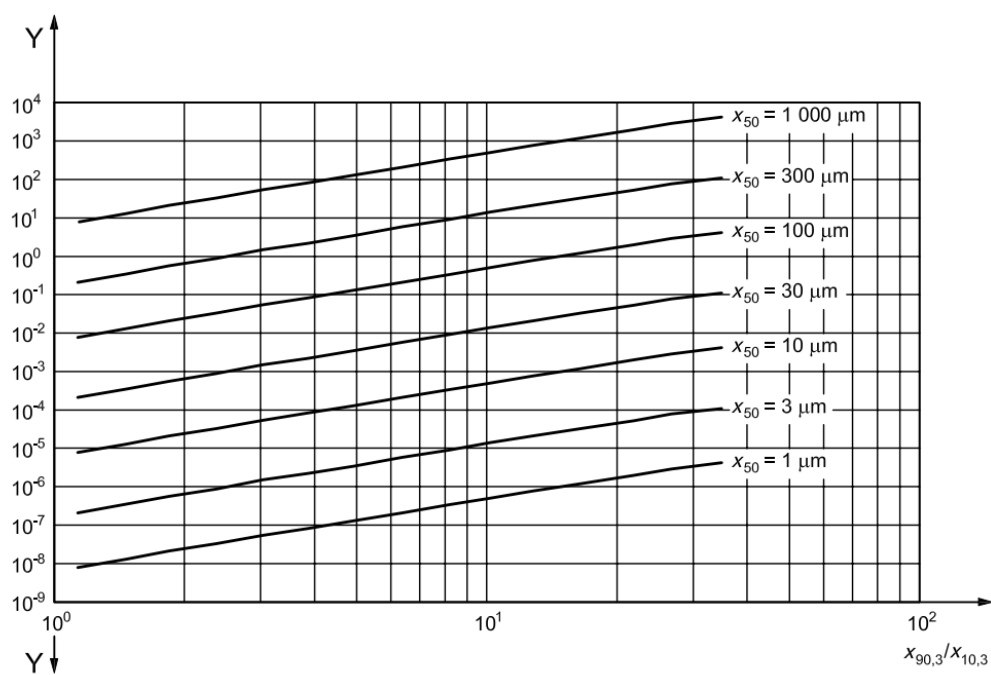
and

$$\text{CV}(n_0) = [100\sqrt{(n_0)}]/n_0 \%$$

$$\text{CV}(n_0) = 100\sqrt{(n_0)} \%$$

Thus, for a precision of 3 percent, the number of particles greater than $x_{90,3}$ will require $n_0 = 1\,111$.

The method of calculating the fundamental error is illustrated in Table 4. The true density ρ of the beads is taken as 2 600 kg/m³. A preliminary PSD analysis of the glass beads is shown in columns A, B and D of the table; columns B and D are normalized to 100 percent.



Key

Y — sample mass required (g)

FIG. 5 GRAPHICAL REPRESENTATION OF SAMPLE MASS REQUIRED FOR A FUNDAMENTAL ERROR OF 3 PERCENT

Table 4 Particle Size Distribution Calculations
(Clause A-3)

Sl No.	A	B	C	D	E	F	G	H	I	J
	x μm	Q_3 % by Mass	$< x >$ μm	dQ_3 % by Mass	$< M_i >$ G	n_i Number in Size Class i per 100 g of Sample	dQ_0 % by Number	Q_0 % by Number	Number if FE (Amount $> x_{90,3}$) to be 3 %	Mass if FE (Amount $> x_{90,3}$) to be 3 % g
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
i)	36	0	39.9	2.2	8.01×10^{-8}	2.75×10^7	17.7	0.0	3.89×10^4	3.11×10^{-3}
ii)	42	2.2	44.9	2.7	1.23×10^{-7}	2.19×10^7	14.1	17.7	3.10×10^4	3.82×10^{-3}
iii)	48	4.9	52.3	6.0	1.95×10^{-7}	3.08×10^7	19.8	31.8	4.36×10^4	8.49×10^{-3}
iv)	57	10.9	62.7	9.0	3.36×10^{-7}	2.68×10^7	17.3	51.6	3.79×10^4	1.27×10^{-3}
v)	69	19.9	74.8	8.8	5.70×10^{-7}	1.54×10^7	9.9	68.9	2.19×10^4	1.25×10^{-3}
vi)	81	28.7	88.2	12.2	9.34×10^{-7}	1.31×10^7	8.4	78.8	1.85×10^4	1.73×10^{-3}
vii)	96	40.9	104.6	14.6	1.56×10^{-6}	9.37×10^6	6.0	87.3	1.33×10^4	2.07×10^{-3}
viii)	114	55.5	124.1	15.0	2.60×10^{-6}	5.77×10^6	3.7	93.3	8.17×10^3	2.12×10^{-3}
ix)	135	70.5	146.5	12.3	4.28×10^{-6}	2.87×10^6	1.9	97.0	4.06×10^3	1.74×10^{-3}
x)	159	82.8	171.1	7.2	6.81×10^{-6}	1.06×10^6	0.7	98.8	1.50×10^3	1.02×10^{-3}
xi)	184	90	188.0	2.3	9.04×10^{-6}	2.54×10^5	0.2	99.5	3.60×10^2	3.25×10^{-3}
xii)	192	92.3	209.2	5.0	1.25×10^{-5}	4.01×10^5	0.3	99.7	5.67×10^2	7.07×10^{-3}
xiii)	228	97.3	248.1	2.7	2.08×10^{-5}	1.30×10^5	0.1	99.9	1.84×10^2	3.82×10^{-3}
xiv)	270	100						100		
xv)	Total			100		1.55×10^8	100.0		2.20×10^5	1.41×10^{-1}
xvi)	Total rows 11 to 13			10.0		7.86×10^5		0.5	1 111	1.41×10^{-2}

NOTE — The values in col C and E to J have been calculated, from which the rounded values have been included in the table. Values calculated directly from those given in col A, B and D may, therefore, show small deviations.

Columns A, B and D give the results of a preliminary analysis. The sum of dQ_3 has been normalized to 100 percent.

The 90th percentile has been added to the data set. The corresponding particle size (cell Axi) has been calculated by linear interpolation from the adjacent size and percentile values:

$$Axi = Ax + (90 - Bx) \times (Axii - Ax)/(Bxii - Bx)$$

Thus, an extra (artificial) size class has been added to the table.

Column B provides the cumulative results for the percentage by mass, while column D shows the differential results. Column C provides the calculated geometric mean size for each size class (the square root of the products of the size class limits).

NOTE — It has been assumed in further calculations that all particles within a size class have the same diameter, this being taken as the geometric mean size ($< x >$). In cases where the classes have a linear scaling, the arithmetic mean size gives a better representation.

In column E, the average mass per particle in each size class has been calculated from the formula $1/6\pi\rho(< x >)^3$ by assuming spherical particles.

Column F gives the number of particles in each size class per 100 g of sample, for example.

$$Fi = (Di)/(100Ei)$$

Cell Fxv gives the total number of particles per 100 g of sample. This sum of Fi to Fxiii permits the calculation of the normalized differential and cumulative number-based size distributions, presented in columns G and H. The value of Fxvi which equals $\text{SUM}(Fxi, Fxii, Fxiii)$, gives the total number of particles larger than $x_{90,3}$ per 100 g of sample. It was used for the normalization of Ixvi to 1 111.

Both mass-based and number-based particle size distributions are illustrated in Fig. 6. Note the large differences between the two distributions.

NOTE — Column H shows that the number fraction $Q_0(x_i)$ smaller than $x_i = x_{90,3}$ is 0.995 (Hxii) while the number fraction larger than $x_i = x_{90,3}$, $[1 - Q_0(x_i)]$, is 0.005. Binomial statistics can be used to give another possibility for calculating variance and standard deviation of $Q_0(x_i)$ [see Equation (1)]:

$$\text{Var}[Q_0(x_i)] = Q_0(x_i) \times [1 - Q_0(x_i)]/n$$

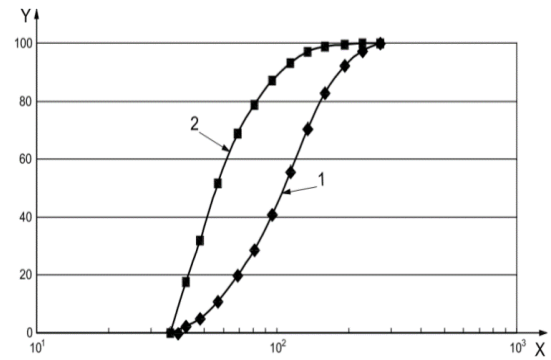
and

$$\sigma[Q_0(x_i)] = \{Q_0(x_i) \times [1 - Q_0(x_i)]/n\}^{0.5}$$

For the total number of particles n given in Ixv), this results for $x_i = 184 \mu\text{m}$ $\sigma(Q_0, 184 \mu\text{m}) = 0.000 15$, which gives a CV of the number fraction of $[1 - Q_0(x_i)] = 0.005$:

$$\text{CV} = 0.000 15 \times 100/0.005 = 3.0 \text{ percent.}$$

It is clear that this is equal to the 3 percent taken as the starting point, using Poisson statistics.



Key

- X Particle size (μm)
- Y Cumulative particle size distribution (%)
- 1 Mass-based
- 2 Number-based

FIG. 6 PRELIMINARY SIZE DISTRIBUTION RESULTS FOR GLASS BEADS

Column I gives the calculated number of particles per size class larger than $x_{90,3}$, if their total were 1 111. The number in I xi) was calculated from Ixi) = $(1\ 111 \times Fxi)/(Fxi)$. The numbers in Ixii) and Ixiii) were calculated similarly. The normalization with Fxvi was used to ensure that $Ixvi = \text{SUM}(Ixi, Ixii, Ixiii) = 1\ 111$. Column J gives the mass of the particles in each size class, obtained by multiplication of the numbers of particles in column I and their corresponding mass (given in column E). Cell Jxvi gives the total mass of the 1 111 particles assumed to be larger than $x_{90,3}$. The total sample mass is given in cell Jxv. The value in cell Jxv can also be calculated by multiplying the summed mass of the particles larger than $x_{90,3}$ given in cell Jxvi by 10, since the total sample mass is, by definition, 10 times the mass above $x_{90,3}$. This leads to a total sample mass of $1.41 \times 10^{-1} = 0.141 \text{ g}$ (141 mg) for the case where an FE (coefficient of variation CV) of 3 percent is taken for the quantity of material larger than the stated characteristic size $x_{90,3}$.

NOTES

1 As stated above, all particles within a size class were assumed to have the same size. Also, linear interpolation was used to divide the number in size class Fxi + Fxii to give a number smaller than $x_{90,3}$ (Fxi) and a number larger than $x_{90,3}$ (Fxii). This will have resulted in a (small) bias. Such bias is not considered significant in providing a robust prediction of the minimum sample mass required to achieve a given precision of FE.

2 The procedure for calculating the data in support of Fig. 5 followed the same basic procedure as for Table 4, the exception being that an analytically generated log-normal distribution was produced for each mean size and distribution width, calculated over 99 size channels.

The calculated minimum sample mass requires an adequate estimate of the width of the particle size distribution. If the preliminary measurement is

executed with a significantly smaller sample mass, then a new measurement should be performed using at least the required minimum mass.

A-4 CONVERSION OF FUNDAMENTAL ERROR IN QUANTITY OF OVERSIZED MATERIAL TO THAT OF CHARACTERISTIC SIZE

Often, the interest is not in the precision of the quantity of material above a stated characteristic size but in the precision of that size itself. This fundamental error in the corresponding characteristic size $x_{90,3}$ can be calculated by division of the standard deviation value calculated above by the slope of the cumulative size distribution at this point of the distribution [see Equation (6)]. The slope of the cumulative size distribution at $x_{90,3}$ was again obtained by linear interpolation:

$$\text{Slope at } x_{90,3} = (B_{xii} - B_x)/(A_{xii} - A_x) = 0.29 \mu\text{m}$$

A 3 percent FE for the amount larger than $x_{90,3}$, which is by definition 10 percent by mass, leads to a standard deviation for Q_3 at $184 \mu\text{m}$ given by:

$$\sigma[Q_3(184 \mu\text{m})] = 3 \times 10/100 = 0.30 \text{ by mass}$$

and for the corresponding standard deviation σ and coefficient of variation CV in the characteristic size $x_{90,3} = 184 \mu\text{m}$ [see Equations (6) and (7)]:

$$\sigma(x_{90,3}) = 0.30/(0.29 \mu\text{m}^{-1}) = 1.04 \mu\text{m}$$

and

$$CV(x_{90,3}) = FE(\text{size}) = 0.57 \text{ percent relative}$$

Thus, for this case, an FE (size) of 0.57 percent

corresponds to 3 percent FE (quantity) at the point $x_{90,3} = 184 \mu\text{m}$.

A-5 EMPIRICAL ESTIMATION OF TOTAL SAMPLING ERROR

The calculation of the total sampling error may be illustrated by a 1 kg batch of glass beads which undergo a degree of segregation. Again, the true density ρ of the beads is taken as $2\,600 \text{ kg/m}^3$.

Five independent samples of 500 mg each were collected for analysis at different locations in the batch of glass beads. For this amount of sample, the numbers of particles larger than the characteristic sizes and their FE were calculated as indicated in A-4 (FE_{calc}). The numbers of particles were: $70\,060 > x_{50,3}$; $4\,238 > x_{90,3}$; $1\,572 > x_{95,3}$; $481 > x_{98,3}$. Note the rapid decrease in the number of particles larger than the size indicated as the characteristic size increases. The corresponding FE_{calc} values are given in row xi of Table 5. A method of analysis was selected that guaranteed good precision for such samples, that is a coefficient of variation for $x_{10,3} - x_{90,3}$ of better than 1 percent and for $x_{90,3} - x_{98,3}$ better than 2 percent.

The results of the analysis are given in Table 5 (rows i to v). The mean values of the measured results were calculated using Equation (8) and the standard deviations (SDs) using Equation (9). The coefficients of variation (CVs) were then obtained by multiplication of the SDs by 100 and division by the mean values [see Equation (7)]. The SD and CV of the mean values were obtained by division of the SD and CV by $\sqrt{N}$ (the square root of the number of measurements).

Table 5 Results of Analysis of Independent 500 mg Samples of Glass Beads

(Clause A-5)

SI No.	Parameters	$x_{10,3}$ μm	$x_{50,3}$ μm	$x_{90,3}$ μm	$x_{95,3}$ μm	$x_{98,3}$ μm	$x_{90,3}/x_{10,3}$
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Result of analysis	53.7	104.5	182.5	208.8	232.2	3.40
ii)		55.2	106.6	178.7	203.6	226.1	3.24
iii)		56.5	109.1	179.9	208.9	231.7	3.18
iv)		57.5	111.1	186.3	215.6	245.6	3.24
v)		54.2	106.1	190.2	220.8	251.3	3.51
vi)	Mean	55.4	107.5	183.5	211.5	237.4	3.31
vii)	SD (pop), μm	1.6	2.6	4.7	6.7	10.6	0.14
viii)	CV (pop), percent	2.9	2.4	2.6	3.2	4.5	4.1
ix)	FE_{calc} percent	—	0.22	0.29	0.43	0.59	—
x)	SD (mean), μm	0.71	1.2	2.1	3.0	4.7	0.06
xi)	CV (mean), percent	1.3	1.1	1.2	1.4	2.0	1.8

Comparison of SI No. (viii) and (ix) in Table 5 shows that the overall sampling precision (expressed by the standard deviations and coefficients of variation of the population) estimated from the five measurements is much larger than the calculated FE. This result indicates that the batch of material is somewhat segregated and/or that the method of sampling was sub-optimal.

A-6 PROCEDURE FOR RELATING PRECISION TO A MINIMUM MASS OF SAMPLE AND MINIMUM NUMBER OF SAMPLES OR SAMPLE INCREMENTS

The procedure for relating a stated precision of a characteristic value of a particle size distribution to a minimum amount of sample and/or to a minimum number of samples or sample increments can now be summarized as follows:

- a) State the characteristic value in the particle size distribution that is proven or expected to represent product quality.
- b) Give a preliminary statement of the precision of this value.
- c) Execute a preliminary measurement of the particle size distribution and convert the volume-based particle size distribution into a number-based distribution.
- d) Check whether Fig. 5 may be helpful for estimating the minimum amount of sample for the stated FE.
- e) Interpolate the stated characteristic value from the measured data, together with the number of oversized particles involved in relation to the stated precision (FE).
- f) Convert, if necessary, the precision stated for the stated characteristic size into a precision for the corresponding number-based quantity.
- g) Calculate the minimum amount of sample related to the stated precision.
- h) If this amount is significantly larger than the amount used in the preliminary measurement of step c, then perform a measurement with at least the newly calculated amount of sample and repeat steps d to g.
- j) Perform measurements with different samples taken from different locations or at different times from the same batch and calculate the precision of the stated characteristic value, using an amount of sample similar to but not less than the calculated minimum amount.
- k) Investigate whether segregation has played an important role in the overall sampling precision. If necessary, improve the method of sampling and start again at step k.
- m) Define the number of samples or sample increments necessary to reach the stated precision for the stated characteristic value.

- n) Calculate the corresponding confidence interval for the measured characteristic value.
- p) If necessary from the point of view of cost and possible from the point of view of quality, reconsider the precision stated in step 'b' and redefine the numbers calculated in step 'n'.

In the above procedure, it has been assumed that sample dispersion and measurement have a minor influence on precision. If there is doubt about this assumption, experimental proof should be provided.

A-7 SIMPLE APPROACH TO THE CALCULATION OF THE FUNDAMENTAL SAMPLING ERROR (FSE) AND MINIMUM MASS REQUIRED FOR A SPECIFIED STANDARD ERROR

This clause provides a method to approximately determine the magnitude (mg/g/kg) of the minimum mass required to meet a specific standard error. The approach approximates the precise calculations described in the document. With a knowledge or assumption of the actual mass utilized in the particle size analysis experiment, the best standard error achievable based solely on the heterogeneity of the material can also be estimated.

The fundamental sampling error (FSE) is one of 8 errors (implies culpability) or variables (implies statistical variation) originally described by Pierre Gy (see Table 6). It represents the smallest possible variation in sample to sample reproducibility based solely on the heterogeneity of the material or distribution involved.

Table 6 Old (Gy) and New Notations (Pitard/Esbensen)

(Clause A-7)

SI No.	Errors	Old Term	New Term
(1)	(2)	(3)	(4)
i)	Heterogeneity fluctuation error	CE	HFE
ii)	Quality fluctuation error	QE	QFE
ii)	Fundamental sampling error	FE	FSE
iv)	Grouping and segregation error	GE	GSE
v)	Increment weighting error	WE	IWE
vi)	Increment delimitation error	DE	IDE
vii)	Increment extraction error	EE	IEE
vii)	Increment preparation errors	PE	IPE

The FSE is identical to the standard error familiar to mathematicians and statisticians. The standard error (SE) of a parameter is the standard deviation (σ , theoretical value) or an estimate of the standard deviation (s , absolute value coming from measurements) of a sampled distribution. If the parameter or the statistic is the mean, it is called the standard error of the mean (SEM). It represents how close the result gets to the ‘true’ mean with repeated samplings or an increased proportion of the actual distribution.

In particle size distribution considerations, the FSE is inversely proportional to the square root of the number of particles present in the distribution or part of the distribution. In the following derivations, no assumption is made of the form of the original particle size distribution; only, that the samples withdrawn are normally distributed (as is the standard case).

$$\text{SEM} \propto 1/\sqrt{n} \text{ or } n \propto 1/\sigma^2$$

For 1 percent SEM it can be shown that:

$$n = 1(0.01)^2 = 10\,000$$

Thus, 10 000 particles in total will be needed to specify the mean to 1 percent SE. The implication is that to specify any other point of the distribution to 1 percent SE, at least 10 000 particles will be needed in the portion of the distribution above that point. The worst-case situation is considered first; specifying the x_{99} point in the distribution to a standard error of 1 percent. This requires 10 000 particles in the $x_{99}+$ part of the distribution. This $x_{99}+$ part represents only 1/100 of the total sample mass of the entire distribution. Thus, only the mass (= volume $\times$ density) of 10 000 particles needs to be calculated at some known or assumed x_{99} point in the distribution and multiplied by the appropriate value to compute the total mass of the distribution. Using the value of the x_{99} point is more convenient than attempting to use a more correct midpoint between the x_{99} and (unknown) x_{100} point. It will slightly underestimate the minimum mass required but calculations show this to be minor.

Making two assumptions, the minimum mass of the portion M_Q can be calculated: the particles are spherical and all particles of the portion have the minimum diameter x_Q .

$$M_Q = n_{Min} \times (\pi/6) \times x_Q^3 \times \rho$$

where

n_{Min} = Minimum number of particles for the specified precision;

$(\pi/6)$ = Shape factor for a sphere;

x_Q = Largest point in the distribution that is to be specified; and

ρ = Density of the particles in the sample.

NOTE — $(\pi/6) \times [x_Q]^3$ is the volume of a single particle of size x_Q and multiplied by the density ρ gives the mass.

This gives the minimum required total mass M_{tot} :

$$M_{tot} = \frac{M_Q}{1 - Q_3(x_Q)} = \frac{n_{Min} \times (\pi/6) \times x_Q^3 \times \rho}{1 - Q_3(x_Q)} = \frac{(\pi/6) \times x_Q^3 \times \rho}{(SE)^2 \times (1 - Q_3(x_Q))}$$

where

$Q_3(x_Q)$ = Volume-based cumulative distribution for the diameter x_Q .

NOTE — All parameters are given in SI units.

M_{tot} , is thus M_Q multiplied by a multiplier, which takes the calculated mass fraction, M_Q to the total mass of sample (for example for the x_{99} point then this is 1 percent of the total sample so the multiplier is 100; for the x_{95} point this multiplier would be 20. Stricter statistical considerations show 98 and 18 respectively for the multiplier for the x_{99} and x_{95} but this does not detract from the broad conclusions presented here).

One simple example can illustrate the calculation, based on an assumed top end parameter x_{99} of 1 000 μm (0.001 m) of particles with a density of 2 500 kg/m^3 . The required standard error SE is 1 percent (0.01) and according to its definition $Q_3(x_{99}) = 99$:

$$M_{tot} = \frac{(\pi/6) \times (0.001 \text{ m})^3 \times 2\,500 \text{ kg/m}^3}{(0.01)^2 \times (1 - 0.99)} = \frac{(\pi/6) \times 2.5 \times 10^{-6} \text{ kg}}{10^{-6}} = 1.3 \text{ kg}$$

The minimum total mass M_{tot} required for an SE of 1 percent for an x_{99} of 1 000 μm is 1.3 kg (see Table 7).

It is easy to spreadsheet such calculations. The example above can be expanded to cover sizes lower and higher in order of magnitude steps (see Table 7).

Table 7 Mass of Test Sample Required to Reach 1 Percent Standard Error for the Upper 1 Percent of a Volume Particle Size Distribution with Particle Density 2.5 g/cm³

(Clause A-7)

Sl No.	x μm	Mass in Top Size Fraction g	Total Mass g	Note
(1)	(2)	(3)	(4)	(5)
i)	1	1 309E-08	131E-06	
ii)	10	1 309E-05	131E-03	~1 mg
iii)	100	1 309E-02	131E+00	~1 g
iv)	1 000	1 309E+01	131E+03	~1 kg
v)	10 000	1 309E+04	131E+06	~1 000 kg
		This is the mass of 10 000 particles assuming spheres	This is where 1 percent of the particles are in the top size band (or 1 percent in last size band – x_{99})	

In summary, it can be stated that at least a test sample or sample increment of 1 g is required to reach a fundamental error (standard deviation) of 1 percent for a $x_{99,3}$ (the 99th percentile of a volume distribution).

Thus, the required sample size mass in mg, g, kg or metric tons is based solely on the top endpoint in the distribution as given in Table 7.

In a more practical case, the calculated sample masses would be reduced by a factor of 250 to meet the 5 percent x_{90} requirement in IS 101 (Part 10/Sec 1)/ISO 13320 (25 fold from 1 percent SE to 5 percent SE and a factor of 10 from x_{99} to x_{90}).

It is possible to reverse the calculation and to calculate the best sample-to-sample precision based solely on the heterogeneity of the material. Therefore, the following parameters need to be known:

- the point in the distribution to specify;
- the density of the material; and
- the amount of sample used in the experiment.

x_{99} , can be taken as an example with sample sizes of 0.1 g/1 g/10 g and with various top end sizes from 1 μm to 2 000 μm , using a low density of 1.5 g/cm³ typical of a pharmaceutical (see Table 8).

Table 8 Standard Error Calculations for the x_{99} Point of the Distribution with Varying Sample Masses (0.1 g/1 g/10 g) and Density 1.5 g/cm³

(Clause A-7)

Sl No.	x μm	$x/10\,000$ (conversion to cm)	x^3 cm^3	ρ g/cm^3	Mass used g	Mass of one particle g	No. particles	Calculated SE %
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	1	0.000 1	1E-12	1.5	0.1	7.853 98E-13	1 273 239 545	0.003
ii)	1	0.000 1	1E-12	1.5	1	7.853 98E-13	12 732 395 447	0.001
iii)	1	0.000 1	1E-12	1.5	10	7.853 98E-13	1.273 24E+11	0.000
iv)	20	0.002	8E-09	1.5	0.1	6.283 19E-09	159 155	0.25
v)	20	0.002	8E-09	1.5	1	6.283 19E-09	1 591 549	0.08
vi)	20	0.002	8E-09	1.5	10	6.283 19E-09	15 915 494	0.03
vii)	50	0.005	1.25E-07	1.5	0.1	9.817 48E-08	10 186	0.99
viii)	50	0.005	1.25E-07	1.5	1	9.817 48E-08	101 859	0.31
ix)	50	0.005	1.25E-07	1.5	10	9.817 48E-08	1 018 592	0.10
x)	100	0.01	0.000 001	1.5	0.1	7.853 98E-07	1 273	2.80
xi)	100	0.01	0.000 001	1.5	1	7.853 98E-07	12 732	0.89
xii)	100	0.01	0.000 001	1.5	10	7.853 98E-07	127 324	0.28
xiii)	200	0.02	0.000 008	1.5	0.1	6.283 19E-06	159	7.93
xiv)	200	0.02	0.000 008	1.5	1	6.283 19E-06	1 592	2.51
xv)	200	0.02	0.000 008	1.5	10	6.283 19E-06	15 915	0.79
xvi)	500	0.05	0.000 125	1.5	0.1	9.817 48E-05	10	31.3
xvii)	500	0.05	0.000 125	1.5	1	9.817 48E-05	102	9.9
xviii)	500	0.05	0.000 125	1.5	10	9.817 48E-05	1 019	3.1
xix)	1 000	0.1	0.001	1.5	0.1	0.000 785 398	1	88.6
xx)	1 000	0.1	0.001	1.5	1	0.000 785 398	13	28.0
xxi)	1 000	0.1	0.001	1.5	10	0.000 785 398	127	8.9
xxii)	1 500	0.15	0.003 375	1.5	0.1	0.002 650 719	0	162.8
xxiii)	1 500	0.15	0.003 375	1.5	1	0.002 650 719	4	51.5
xxiv)	1 500	0.15	0.003 375	1.5	10	0.002 650 719	38	16.3
xxv)	2 000	0.2	0.008	1.5	0.1	0.006 283 185	0	250.7
xxvi)	2 000	0.2	0.008	1.5	1	0.006 283 185	2	79.3
xxvii)	2 000	0.2	0.008	1.5	10	0.006 283 185	16	25.1

At the larger sizes ($> 100\ \mu\text{m}$ or so), the minimum variation is substantial if sample masses are kept below ≈ 10 g. This has implications for particle size distributions obtained by such techniques as electron microscopy.

At 20 mg total sample mass, the variation on the x_{99} (the number of calculated particles is in a mass of 0.000 2 g for the x_{99}) is given in Table 9.

Table 9 Standard Error Calculations for the x_{99} Point in the Distribution with Sample Mass 20 mg and Density 1.5 g/cm³*(Clause A-7)*

Sl No.	x µm	ρ g/cm³	Mass Used g	Mass of One Particle g	No. Particles	Calculated SE %
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	1	1.5	0.02	7.853 98E-13	254 647 909	0.01
ii)	20	1.5	0.02	6.283 19E-09	31 831	0.56
iii)	50	1.5	0.02	9.817 48E-08	2 037	2.22
iv)	100	1.5	0.02	7.853 98E-07	255	6.27
v)	200	1.5	0.02	6.283 19E-06	32	17.72
vi)	500	1.5	0.02	9.817 48E-05	2	70.06
vii)	1 000	1.5	0.02	0.000 785 398	0	198.17
viii)	1 500	1.5	0.02	0.002 650 719	0	364.05
ix)	2 000	1.5	0.02	0.006 650185	0	560.50

NOTE — The above calculations provide the most suitable sample-to-sample variation based solely on the heterogeneity of the material. All other errors or variation will add to this minimum.

ANNEX B

(Clause 8)

CALCULATION OF VARIANCES AT DIFFERENT STAGES IN THE SAMPLING SEQUENCE

B-1 GENERAL

The variances at the different stages in the sampling sequence can be estimated by an experimental method covering either the full sequence or part of the sequence.

B-2 FULL SEQUENCE

In the full sequence, all sub-samples taken are further sampled or analysed. The symbols x_i, x_{ij}, x_{ijk} denote the experimental values of the random variable x at the different stages of sampling and

size analysis (see Fig. 7). The notation x_{ij**} is used for the mean over k , x_{i**} is used for the mean over j and k , and x_{**} is used for the mean over i, j and k . The number of primary sample increments (stage denoted by ξ_i) is denoted by n_1 , the number of secondary sub-samples (stage denoted by ψ_{ij}) is denoted by n_2 and the number of size analyses (stage denoted by ζ_{ijk}) from each sub-sample is denoted by n_3 . Using this notation, the sum of the squares of the measurements can be given as:

$$\sum_{ijk} (x_{ijk} - x_{***})^2 = \sum_{ijk} (x_{ijk} - x_{ij*})^2 + n_3 \sum_{ij} (x_{ij*} - x_{i**})^2 + n_2 n_3 \sum_i (x_{i**} - x_{***})^2 \quad \dots (12)$$

or, in a shorter notation,

$$\sum_{ijk} (x_{ijk} - x_{***})^2 = s_0 + s_1 + s_2 \quad \dots (13)$$

The sums of squares represent estimates of the variations in the different phases of the error hierarchy as given in Table 3.

Table 3 The Estimates that the Sums of Squares Represent

(Clause B-2)

Sl No.	Sum of Squares	Number of Degrees of Freedom	Represents an Estimate of
(1)	(2)	(3)	(4)
i)	$s_0 = \sum_{ijk} (x_{ijk} - x_{ij*})^2$	$n_1 n_2 (n_3 - 1)$	$n_1 n_2 (n_3 - 1) \sigma_0^2$
ii)	$s_1 = n_3 \sum_{ij} (x_{ij*} - x_{i**})^2$	$n_1 (n_2 - 1)$	$n_1 (n_2 - 1) (n_3 \sigma_1^2 + \sigma_0^2)$
iii)	$s_2 = n_2 n_3 \sum_i (x_{i**} - x_{***})^2$	$(n_1 - 1)$	$(n_1 - 1) (n_2 n_3 \sigma_2^2 + n_3 \sigma_1^2 \sigma_0^2)$

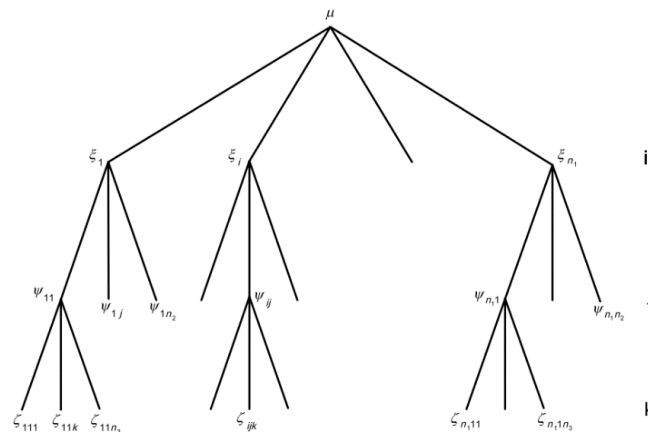


FIG. 7 SAMPLING SEQUENCE ERRORS

[For meanings of symbols (see B-2)]

Using Table 3, we can obtain the equations used for testing the statistical significance of the variances of the different stages of sample preparation and division.

For the statistical assumption that $\sigma_1 = 0$, the test equation is

$$\frac{\frac{s_1}{[n_1(n_2-1)]}}{\frac{s_0}{[n_1 n_2 (n_3-1)]}} = F_{\phi_1 \phi_2}$$

$$= F_{[n_1(n_2-1)], [n_1 n_2 (n_3-1)]} \quad \dots (14)$$

where

F_{ϕ_1, ϕ_2} = Standard F -distribution value with ϕ_1 and ϕ_2 degrees of freedom.

In a similar manner, a test function can be formulated for the statistical assumption that $\sigma_2 = 0; \sigma_1 = 0$. If this assumption cannot be accepted, then

$$\frac{\frac{s_2}{n_1-1}}{\frac{s_1}{[n_1(n_2-1)]}} = F_{\phi_1 \phi_2}$$

$$= F_{(n_1-1), [n_1(n_2-1)]} \quad \dots (15)$$

If the statistical assumption that $\sigma_2 = 0; \sigma_1 = 0$ can be accepted, the test function is

$$\frac{s_2/(n_1-1)}{(s_0 + s_1)/[n_1(n_2 n_3 - 1)]} = F_{\phi_1 \phi_2}$$

$$= F_{(n_1-1), [n_1(n_2 n_3 - 1)]} \quad \dots (16)$$

The latter equation should be used if the assumption of test function (14) is accepted.

If neither the assumption $\sigma_2 = 0$ nor the assumption $\sigma_1 = 0$ can be accepted, then estimates for the variances of different sample treatment tasks can be evaluated by solving the equations in Table 3.

Variances should not be estimated if the estimates prove to be without significance.

B-3 PARTIAL SEQUENCE

In the case of a partial sequence, only a selected number of sub-samples are treated further. Let the number of sample divisions be denoted as K_i for sampling stage i and the number of sub-samples taken be n_i for stage i .

The mean value $\bar{x}$ of the random variable x is then (see Fig. 8)

$$\bar{x} = \frac{1}{n_1 n_2 n_3} \sum_{i=1}^{n_1} \sum_{j=1}^{n_2} \sum_{k=1}^{n_3} x_{ijk} \quad \dots (17)$$

The estimates for the variances for the stages are:

$$s_3^2 = \frac{1}{n_1 n_2 (n_3-1)} \sum_{i=1}^{n_1} \sum_{j=1}^{n_2} \sum_{k=1}^{n_3} (x_{ijk} - \bar{x}_{ij})^2 \quad \dots (18)$$

$$s_2^2 = \frac{1}{n_1 (n_2-1)} \sum_{i=1}^{n_1} \sum_{j=1}^{n_2} (x_{ij} - \bar{x}_i)^2 - \frac{s_3^2}{n_3} \quad \dots (19)$$

and

$$s_1^2 = \frac{1}{(n_1-1)} \sum_{i=1}^{n_1} (x_i - \bar{x})^2 - \frac{s_2^2}{n_2} - \frac{s_3^2}{n_2 n_3} \quad \dots (20)$$

In these equations

$$x_{ij} = \frac{1}{n_3} \sum_{k=1}^{n_3} x_{ijk} \quad \dots (21)$$

and

$$x_i = \frac{1}{n_2} \sum_{j=1}^{n_2} x_{ij} \quad \dots (22)$$

The variance for real sequential sampling is:

$$\sigma_{po}^2 = \frac{K_1 - n_1}{K_1 - 1} \frac{s_1^2}{n_1} + \frac{1}{n_1} \frac{K_2 - n_2}{K_2 - 1} \frac{s_2^2}{n_2} + \frac{1}{n_1 n_2} \frac{K_3 - n_3}{K_3 - 1} \frac{s_3^2}{n_3} \quad \dots (23)$$

The test equations are the same as in full sequence calculations.

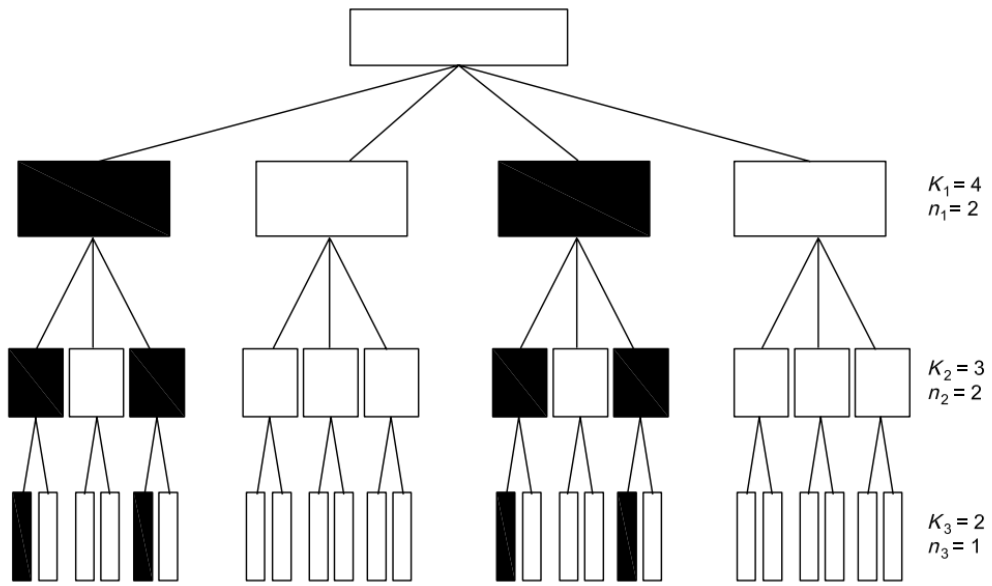


FIG. 8 PARTIAL SAMPLING SEQUENCE – EXAMPLE IN THREE STAGES

ANNEX C

(Foreword)

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